

1000 15631-15631  
JC10 Rec'd PCT/PTO 27 DEC 2001

FORM PTO-1390  
OFFICE  
(REV. 11-2000)

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK

ATTORNEY'S DOCKET NUMBER

**TRANSMITTAL LETTER TO THE UNITED STATES  
DESIGNATED/ELECTED OFFICE (DO/EO/US)  
CONCERNING A FILING UNDER 35 U.S.C. 371**

45900-000714/US

U.S. APPLICATION NO. (If known, see 37 CFR 1.5)

**10/019631**

INTERNATIONAL APPLICATION NO.

INTERNATIONAL FILING DATE

PRIORITY DATE CLAIMED

PCT/DK00/00348

June 28, 2000

June 28, 1999

TITLE OF INVENTION

NANOMETER-SCALE MODULATION

APPLICANT(S) FOR DO/EO/US

Hasin F. Grey et al

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39 (1).
4. ☒ The US has been elected by the expiration of 19 months from the priority date (Article 31).
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
  - a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau). WO 00/
  - b. ☒ has been transmitted by the International Bureau.
  - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☒ An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)).
  - a. ☒ is transmitted herewith.
  - b. ☐ has been previously submitted under 35 U.S.C. 154(d)(4)
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3)).
  - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
  - b. ☒ have been transmitted by the International Bureau.
  - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
  - d. ☐ have not been made and will not be made.
8. ☐ An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☐ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ An English language translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 20. below concern document(s) or information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98-1449 and International Search Report (PCT/ISA/210) with two (2) references.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A **FIRST** preliminary amendment.
14. ☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
15. ☐ A substitute specification.
16. ☐ A change of power of attorney and/or address letter.
17. ☐ A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821-1.825.
18. ☐ A second copy of the published international application under 35 U.S.C. 154(d)(4).
19. ☐ A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).
20. ☐ Other items or information:

U.S. APPLICATION NO. (known) <b>10/019631</b> <small>NEW</small>		INTERNATIONAL APPLICATION NO.		ATTORNEY'S DOCKET NUMBER 45900-000714/US	
21. <input checked="" type="checkbox"/> The following fees are submitted <b>BASIC NATIONAL FEE (37 CFR 1.492(a)(1)-(5):</b> Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO. . . . . <b>\$1,040.00</b>  International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO . . . . <b>\$890.00</b>  International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO. . . . . <b>\$710.00</b>  International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4) . . . . . <b>\$690.00</b>  International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4). . . . . <b>\$100.00</b> <b>ENTER APPROPRIATE BASIC FEE AMOUNT =</b>				<b>CALCULATIONS      PTO USE ONLY</b>	
Surcharge of <b>\$130.00</b> for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input checked="" type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(e)).				\$	890.00
				\$	130
CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE		
Total Claims	36 - 20 =	16	X <b>\$18.00</b>	\$	288
Independent Claims	2 - 3 =	0	X <b>\$80.00</b>	\$	0
MULTIPLE DEPENDENT CLAIM(S) (if applicable)      None			+ <b>\$270.00</b>	\$	0
<b>TOTAL OF ABOVE CALCULATIONS =</b>				\$	1020
<input checked="" type="checkbox"/> Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above are reduced by 1/2.				\$	510
<b>SUBTOTAL =</b>				\$	510
Processing fee of <b>\$130.00</b> for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f)).				\$	0
<b>TOTAL NATIONAL FEE =</b>				\$	510
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). <b>\$40.00</b> per property +				\$	
<b>TOTAL FEES ENCLOSED =</b>				\$	510
				Amount to be:	
				refunded	\$
				charged	\$

a. ☐ A check in the amount of \$ \_\_\_\_\_ to cover the above fees is enclosed.

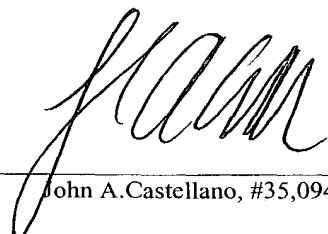
b. ☒ Please charge my Deposit Account. No. 08-0750 in the amount of \$510.00 to cover the above fees.  
A triplicate copy of this sheet is enclosed.

c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any  
overpayment to Deposit Account No. 08-0750.

**NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.**

Send all correspondence to  
**Harness, Dickey & Pierce, P.L.C – Customer No. 30596**  
**Post Office Box 8910**  
**Reston, Virginia 20195**

**Date: December 27, 2001**

By  \_\_\_\_\_  
 John A. Castellano, #35,094

/kna

WO 01/00522 A3



**(84) Designated States (regional):** ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

**(88) Date of publication of the international search report:**  
3 May 2001

*For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette*

**Published:**

— *With international search report.*

10019510/019631  
531 Rec'd PCT/ 27 DEC 2001  
PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application No.: To be assigned  
Filing Date: December 27, 2001  
Applicant: Hasin F. Grey et al.  
Group Art Unit: To be assigned  
Examiner: To be assigned  
Title: NANOMETER-SCALE MODULATION  
Attorney Docket: 45900-000714

---

Box Non-Fee Amendment;  
Commissioner of Patents and Trademarks  
Washington, D.C. 20231

PRELIMINARY AMENDMENT

Sir:

Applicant submits this Preliminary Amendment to the application filed herewith, for consideration prior to the calculation of the filing fee, as follows:

This application is the National Phase under 35 USC §371 of PCT International Application No. PCT/DK00/00348 which has an International filing date of June 28, 2000, which designated the United States of America the entire contents of which are hereby incorporated by reference.

### IN THE CLAIMS

Please amend the claims in accordance with the following rewritten claims in clean form. Applicant includes herewith an Attachment for Claim Amendments showing a marked up version of each amended claim.

Please cancel Claims 1-29 without prejudice or disclaimer of the subject matter contained therein.

Please add the following new claims:

30. [Added] A method for providing a substantially periodic pattern, said method comprising the steps of:

- providing a first group of crystalline elements being formed by the same material and having a predetermined first crystal axis,
- providing a second group of crystalline elements being formed by the same material and having a predetermined second crystal axis being different from the first crystal axis, and
- bringing the first and second group of crystalline elements into contact with each other.

31. [Added] A method according to claim 30, wherein the material forming the first group of crystalline elements comprises a semiconductor material.

32. [Added] A method according to claim 31, wherein the semiconductor material is silicon or gallium arsenide.

33. [Added] A method according to claim 31, wherein the material forming the second group of crystalline elements comprises a semiconductor material

34. [Added] A method according to claim 33, wherein the semiconductor material is silicon or gallium arsenide.

35. [Added] A method according to claim 30, wherein the materials forming the first and second group of crystalline elements comprise an insulator material.

36. [Added] A method according to claim 35, wherein the insulator material is diamond or sapphire.

37. [Added] A method according to claim 30, wherein the first crystal axis differs from the second crystal axis by a twist angle  $\theta$ , wherein  $\theta$  is within the range 1-5°.

38. [Added] A method according to claim 30, wherein the first crystal axis differs from the second crystal axis by a tilt angle  $\varphi$ , wherein  $\varphi$  is within the range 1-5°.

39. [Added] An article comprising:

- a first group of crystalline elements being formed by the same material and having a predetermined first crystal axis,
- a second group of crystalline elements being formed by the same material and having a predetermined second crystal axis being different from the first crystal axis,

wherein the first and second group of crystalline elements are adjacently positioned so as to form an interface region between the first and second group of crystalline elements, at least part of said interface region defining a substantially periodic pattern extending in at least one direction.

40. [Added] An article according to claim 39, wherein the material forming the first and second groups of crystalline elements comprise a semiconductor material.

41. [Added] An article according to claim 40, wherein the semiconductor material is silicon or gallium arsenide.

42. [Added] An article according to claim 39, wherein the materials forming the first and second group of crystalline elements comprise an insulator material.

43. [Added] An article according to claim 42, wherein the insulator material is diamond or sapphire.

44. [Added] An article according to claim 39, wherein the first crystal axis differs from the second crystal axis by a twist angle  $\theta$ , wherein  $\theta$  is within the range 1-5°.

45. [Added] An article according to claim 39, wherein the first crystal axis differs from the second crystal axis by a tilt angle  $\varphi$ , wherein  $\varphi$  is within the range 1-5°.

46. [Added] A laser comprising:

- an article according to claim 39, the article further comprising:

- a material or a material system so as to form an array of quantum dots and/or quantum wires at or near the interface region, wherein the further material or material system distributes according to the substantially periodic pattern, and

- means for providing a pump signal for pumping the material or material system so as to emit electromagnetic radiation.

47. [Added] A laser according to claim 46, wherein the material or material system forming the array of quantum dots and/or quantum wires comprises a semiconductor material.

48. [Added] A laser according to claim 47, wherein semiconductor material is gallium, arsenide, indium or any combination thereof.

49. [Added] A laser according to claim 46, wherein the material or material system forming the array of quantum dots and/or quantum wires has a thickness smaller than 50 nm.

50. [Added] A laser according to claim 46, wherein the material or material system forming the array of quantum dots and/or quantum wires is overgrown with an additional material.

51. [Added] A laser according to claim 50, wherein the additional material is silicon or gallium arsenide.

52. [Added] A laser according to claim 46, wherein the pump signal comprises electromagnetic radiation.

53. [Added] A laser according to claim 52, wherein the electromagnetic radiation is in the radio frequency, visible or near-infrared range.

54. [Added] A laser according to claim 46, wherein the pump signal comprises a direct and/or alternating electric current.

55. [Added] A laser according to claim 46, wherein the emitted electromagnetic radiation is in the visible or near-infrared range.

56. [Added] An object for calibrating an instrument, said object comprising:

- an article according to claim 39, wherein the substantially periodic pattern is transferred to a surface of the article.



- an article according to claim 39, wherein the substantially periodic pattern is transferred to a surface of the article, said transferred substantially periodic pattern being adapted to hold a plurality of magnetic structures so as to form a plurality of magnetic domains.

63. [Added] An object according to claim 62, wherein the plurality of magnetic structures is selected from the group consisting of iron, cobalt, chromium or any combination thereof.

64. [Added] An object according to claim 62, wherein the plurality of magnetic structures are arranged according to the substantially periodic structure.

65. [Added] An object according to claim 62, wherein the plurality of magnetic structures are overgrown with a non-magnetic material.

**REMARKS**

Claims 1-29 were cancelled and claims 30-65 are now pending in the application. The amendments to the claims contained herein are of equivalent scope as originally filed and, thus, are not a narrowing amendment. It is submitted that this Amendment places the application in suitable condition for allowance; notice of which is requested. If the Examiner believes that personal communication will expedite prosecution of this application, the Examiner is invited to telephone the undersigned at (703) 390-3030.

Respectfully submitted,

Dated: 12/27/01

By: 

John A. Castellano  
Reg. No. 35,094

HARNESS, DICKEY & PIERCE, P.L.C.  
P.O. Box 8910  
Reston, Virginia 20195  
(703) 390-3030

**ATTACHMENT FOR CLAIM AMENDMENTS**

The following is a marked up version of each amended claim in which underlines indicates insertions and brackets indicate deletions.

30. [ A method for providing a substantially periodic pattern, said method comprising the steps of:

- providing a first group of crystalline elements being formed by the same material and having a predetermined first crystal axis,
- providing a second group of crystalline elements being formed by the same material and having a predetermined second crystal axis being different from the first crystal axis, and
- bringing the first and second group of crystalline elements into contact with each other.]

31. [A method according to claim 30, wherein the material forming the first group of crystalline elements comprises a semiconductor material.]

32. [A method according to claim 31, wherein the semiconductor material is silicon or gallium arsenide.]

33. [A method according to claim 31, wherein the material forming the second group of crystalline elements comprises a semiconductor material]

34. [A method according to claim 33, wherein the semiconductor material is silicon or gallium arsenide.]

35. [A method according to claim 30, wherein the materials forming the first and second group of crystalline elements comprise an insulator material.]

36. [A method according to claim 35, wherein the insulator material is diamond or sapphire.]

37. [A method according to claim 30, wherein the first crystal axis differs from the second crystal axis by a twist angle  $\theta$ , wherein  $\theta$  is within the range 1-5°.]

38. [A method according to claim 30, wherein the first crystal axis differs from the second crystal axis by a tilt angle  $\varphi$ , wherein  $\varphi$  is within the range 1-5°.]

39. [An article comprising:

- a first group of crystalline elements being formed by the same material and having a predetermined first crystal axis,

- a second group of crystalline elements being formed by the same material and having a predetermined second crystal axis being different from the first crystal axis,

wherein the first and second group of crystalline elements are adjacently positioned so as to form an interface region between the first and second group of crystalline elements, at least part of said interface region defining a substantially periodic pattern extending in at least one direction.]

40. [An article according to claim 39, wherein the material forming the first and second groups of crystalline elements comprise a semiconductor material.]

41. [An article according to claim 40, wherein the semiconductor material is silicon or gallium arsenide.]

42. [An article according to claim 39, wherein the materials forming the first and second group of crystalline elements comprise an insulator material.]

43. [An article according to claim 42, wherein the insulator material is diamond or sapphire.]

44. [An article according to claim 39, wherein the first crystal axis differs from the second crystal axis by a twist angle  $\theta$ , wherein  $\theta$  is within the range  $1-5^\circ$ .]

45. [An article according to claim 39, wherein the first crystal axis differs from the second crystal axis by a tilt angle  $\phi$ , wherein  $\phi$  is within the range  $1-5^\circ$ .]

46. [A laser comprising:

- an article according to claim 39, the article further comprising:
  - a material or a material system so as to form an array of quantum dots and/or quantum wires at or near the interface region, wherein the further material or material system distributes according to the substantially periodic pattern, and
  - means for providing a pump signal for pumping the material or material system so as to emit electromagnetic radiation.]

47. [A laser according to claim 46, wherein the material or material system forming the array of quantum dots and/or quantum wires comprises a semiconductor material.]

48. [A laser according to claim 47, wherein semiconductor material is gallium, arsenide, indium or any combination thereof.]

57. [A method of fabricating the object according to claim 56, wherein the step of transferring the substantially periodic pattern to the surface comprises a step of removing at least part of the first or second group of crystalline elements by etching.]

[illegible]

58. [A method of fabricating the object according to claim 56, wherein the step of transferring the substantially periodic pattern to the surface comprises a step of removing at least part of the first or second group of crystalline elements by chemical-mechanical polishing.]

59. [An object according to claim 56, wherein the transferred substantially periodic pattern is adapted to hold an additional material.]

60. [An object according to claim 59, wherein the additional material is a metal.]

61. [An element for splitting an incoming beam into one or more outgoing beams, said element comprising:

- an article according to claim 39, wherein the incoming beam is incident on at least part of the substantially periodic pattern, said incoming beam having a first propagating direction, and wherein the one or more outgoing beams are reflected or transmitted by at least part of the substantially periodic pattern in one or more propagation directions being different from the first propagation direction.]

62. [An object for magnetically storing information, the said object comprising

- an article according to claim 39, wherein the substantially periodic pattern is transferred to a surface of the article, said transferred substantially periodic pattern being adapted to hold a plurality of magnetic structures so as to form a plurality of magnetic domains.]

63. [An object according to claim 62, wherein the plurality of magnetic structures is selected from the group consisting of iron, cobalt, chromium or any combination thereof.]



64. [An object according to claim 62, wherein the plurality of magnetic structures are arranged according to the substantially periodic structure. ]

65. [An object according to claim 62, wherein the plurality of magnetic structures are overgrown with a non-magnetic material.]

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization  
International Bureau



(43) International Publication Date  
4 January 2001 (04.01.2001)

PCT

(10) International Publication Number  
**WO 01/00522 A3**

(51) International Patent Classification<sup>7</sup>: **B82B 1/00.**  
H01L 21/02, 21/18

(21) International Application Number: PCT/DK00/00348

(22) International Filing Date: 28 June 2000 (28.06.2000)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:  
PA 1999 00918 28 June 1999 (28.06.1999) DK

(71) Applicant (for all designated States except US):  
**MIKROELEKTRONIK CENTRET (MIC)** [DK/DK];  
The Technical University of Denmark (DTU), Building  
345 East, DK-2800 Lyngby (DK).

(72) Inventors; and

(75) Inventors/Applicants (for US only): **GREY, Hasin,**  
**François de Charmoy** [CA/DK]; Boldhusgade 4, 1,

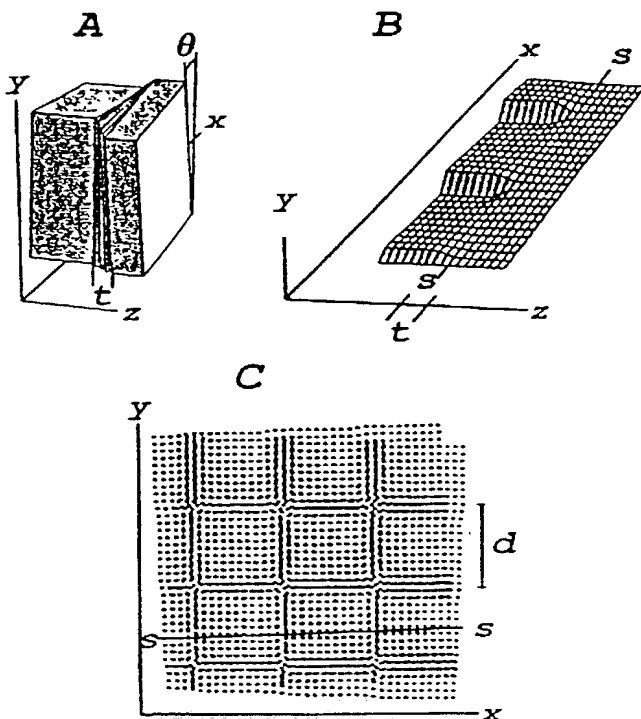
DK-1602 Copenhagen K (DK). **FEIDENHANS'L,**  
**Robert, Krarup** [DK/DK]; Møllehusvej 64, DK-4000  
Roskilde (DK). **VEDDE, Jan** [DK/DK]; J.N. Vinthersvej  
5, DK-3460 Birkerød (DK). **NIELSEN, Mourits**  
[DK/DK]; Naskovvej 14, Veddelev, DK-4000 Roskilde  
(DK) **HOWES, Paul, Bedford** [GB/GB]; 6 Dean Road,  
Hinckley, Leicestershire LE10 1LG (GB).

(74) Agent: **PLOUGMANN, VINGTOFT & PARTNERS**  
A/S; Sankt Annæ Plads 11. P.O. Box 3007, DK-1021  
Copenhagen K (DK).

(81) Designated States (national): AE, AG, AL, AM, AT, AT  
(utility model), AU, AZ, BA, BB, BG, BR, BY, CA, CH,  
CN, CR, CU, CZ, CZ (utility model), DE, DE (utility  
model), DK, DK (utility model), DM, DZ, EE, EE (utility  
model), ES, FI, FI (utility model), GB, GD, GE, GH, GM,  
HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KR (utility  
model), KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG,  
MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE,  
SG, SI, SK, SK (utility model), SL, TJ, TM, TR, TT, TZ,  
UA, UG, US, UZ, VN, YU, ZA, ZW.

[Continued on next page]

(54) Title: NANOMETER-SCALE MODULATION



(57) Abstract: A new method to artificially modulate a crystal lattice is disclosed, where the modulation has a controlled periodicity and thickness in the range of one to several hundred nanometers. The present invention relates to the fabrication of periodically strained crystal lattices where the period and thickness of the modulated region is controlled by bonding two crystal wafers at a specified twist angle. Two polished and clean crystal wafers are placed in intimate contact at a specified twist angle and, if necessary, heated to obtain bonding between the two wafers. The two crystal lattices modulate each other, resulting in a modulation near the interface between the crystal with a periodicity that is different from that of the crystal lattices. This periodic modulation affects the electronic and structural properties of the two crystals in the vicinity of the interface. The modulation of the electronic properties leads to the formation of a highly regular quantum dot or quantum wire structures of controlled period near the crystal interface, with applications in electronics, optoelectronics and magnetic devices. The modulation of the crystal structure can be exploited for the formation of metrological standards in the 1-100 nanometer range, or gratings for diffractive optic elements with grating periods in the same range. By transferring the periodic modulation to the free surface of a crystal, it can be used as a template for further growth of periodically modulated crystalline layers by evaporation techniques.

WO 01/00522 A3

## NANOMETER-SCALE MODULATION

## FIELD OF THE INVENTION

5 The present invention relates to techniques for fabricating artificial periodic structures in crystals and at crystal interfaces and surfaces. In particular the present invention relates to techniques for fabricating nanometer-scale periodic strain patterns near the interface between two crystals, where the strain extends some distance away from the interface.

10

The present invention further relates to the use of such strain patterns to generate a regular lattice of quantum dots or quantum wires for applications in electronic, optoelectronic and magnetic device fabrication. The present invention even further relates to the use of such strain patterns to generate a regular nanoscale lattice of controlled  
15 period at or near the free surface of a crystal for applications in diffractive optical systems, metrology and crystal growth.

## BACKGROUND OF THE INVENTION

20 The most common way of making artificial periodic structures with nanometer-scale periods on crystals is by lithographic techniques such as photolithography and electron beam lithography. Such periodic structures are of technological interest for a variety of applications in electronics and optoelectronics, especially on semiconductor crystals, in storage systems, especially magnetic storage, using magnetic materials,  
25 and in the production of diffraction grating, typically made of metallic materials.

However, lithographic techniques have serious limitations. Optical lithography has limitations mainly in terms of the smallest feature sizes that can be generated, which due to diffraction effects are not smaller than half the wavelength of the light used,  
30 and in practice are considerably larger than this diffraction limit. For example, grating like modulation of GaAs/AlGaAs quantum wells has been achieved by use of an interference pattern from a laser, but the period of the fringes was limited to 2  $\mu\text{m}$  using 532 nm laser light.

Electron beam lithography can be used to make features with smaller periods. However, the technology is slow if large areas on a surface must be patterned. And the regularity of the periodic pattern can be limited by aberrations of the electron optics. For example, Messica et al. describe an array of 25 metallic wires with a period of  
5 100 nm formed by e-beam lithography.

In US patent US 5,532,510 a method is proposed for making periodic strain patterns by generating 10 nanometer-wide apertures by electron or ion beam lithography, but the issues of pattern regularity and processing speed are not addressed.  
10

An alternative method for producing large scale, highly regular periodic patterns near crystal surfaces has been presented by Rocke et al., and involves the use of surface acoustic waves to modulate the strain of the crystal.

15 It is a disadvantage that this method is limited by the wavelength of the surface acoustic waves that can be generated (typically several microns), and is in any case cumbersome, as it requires external power to maintain the modulation.

Low-dimensional systems in optoelectronics, and in particular towards the fabrication  
20 of so-called quantum wires and quantum dots have recently gained a lot of interest. Quantum wires and dots have dimensions in the range of 1-100 nm to provide sufficient confinement of electrons so that the desired quantisation effects are detectable at room temperature. Quantum wires induced by periodic strain have been made by the technique of cleaved-edge overgrowth.

25

It is a disadvantage that the above-mentioned technique is based on growing alternating layers of different materials by molecular beam epitaxy, which is rather cumbersome for the production of structures with more than a few periods.

30 It is a further disadvantage that there is no obvious way to extend the above-mentioned technique to fabricate regular arrays of quantum dot structures.

There is an extensive literature on the formation of arrays of quantum dot structures by so-called Stranski-Krastanov growth, whereby an overgrown material spontane-

ously forms nanometer scale islands on a surface, rather than growing in a layer by layer fashion. For example, Sopanen et al. have used the elastic strain induced by such islands to modulate the electronic structure of a semiconductor layer buried beneath the surface. The limitations of this technique for forming regular periodic structures are the lack of control over the regularity and periodicity of the array of islands. Multilayer techniques may improve the uniformity of the size and spacing of the islands, as described by Tersoff et al. and in US Patent US 5,614,435. However, these techniques result in something that is still very far from the perfection of a single crystal lattice. Another approach to making regimented arrays using electrochemical techniques to form arrays of etch pits on which the islands nucleate is described in US 5,747,180. But again, regularity is only achieved in a statistical sense.

Bonding of crystal wafers has been used to form substrates that are suitable for epitaxial growth of other materials, as described in US Patent US 5,294,808. However, US Patent 5,294,808 pays no attention to the use of any periodic structure generated at the bonded interface, and indeed depends on a lack of strain at the interface.

The strain at a bonded interface of two gold crystals has been investigated by Sass et al. and Taylor et al. However, these authors do not consider the possibility that this strain may effect the electronic properties of the crystals, or the possibility of using this strain at the surface of a crystal to modulate materials grown on top of the crystal.

From the above discussion, it appears that no technique is currently available for fabricating highly regular periodic structures over large areas of a crystal with periodicities in the range 1-100 nm. Furthermore, no attention has been drawn towards the used of such highly periodic structures.

It is an object of the present invention to utilise the periodically induced strain extending from an interface to modify the electronic and/or mechanical structure of a material in a controlled way so as to achieve a desired and technologically useful result.

It is a further object of the present invention to provide a technique for fabrication of highly regular periodic strain modulation in a crystal with periods in the range 1-100 nm.

- 5 It is a still further object of the present invention to provide a substantially periodic structure formed over large areas of a crystal interface by bonding of two crystal wafers.

It is an advantage of the present invention that the periodic strain structure can be  
10 fabricated to extend some significant distance away from the interface, of order 1-10 nm, and that this depth is controllable.

It is a further advantage of the present invention that the regularity of the fabricated periodic structure, which is given by the regularity of the crystal lattices on either  
15 side of the interface, may be extremely high.

#### SUMMARY OF THE INVENTION

The above-mentioned objects are complied with by providing, in a first aspect, a  
20 method for providing a substantially periodic pattern, said method comprising the steps of:

- providing a first group of crystalline elements being formed by the same material and having a predetermined first crystal axis,  
25
- providing a second group of crystalline elements being formed by the same material and having a predetermined second crystal axis being different from the first crystal axis, and
- 30 - bringing the first and second group of crystalline elements into contact with each other.

In a second aspect, the present invention relates to an article comprising:

- a first group of crystalline elements being formed by the same material and having a predetermined first crystal axis,
- 5       - a second group of crystalline elements being formed by the same material and having a predetermined second crystal axis being different from the first crystal axis,
- 10   wherein the first and second group of crystalline elements are adjacently positioned so as to form an interface region between the first and second group of crystalline elements, at least part of said interface region defining a substantially periodic pattern extending in at least one direction.
- 15   The material forming the first group of crystalline elements may comprise a semiconductor material, such as silicon or gallium arsenide. Similarly, the material forming the second group of crystalline elements may comprise a semiconductor material, such as silicon or gallium arsenide. Alternatively, the materials forming the first and second group of crystalline elements may comprise an insulator material, such as
- 20   diamond or sapphire.

The orientation of the crystal axes of the two wafers may differ in two different ways. There may be a twist angle between the crystal axes such that the in-plane axes of first crystal axis are rotated an angle  $\theta$  relative to second crystal axis at the

25   time of bonding.

There may also be a tilt angle  $\phi$  the crystal axes. This can be achieved by cutting the surface of one of the crystals at a tilt angle  $\phi$  to the main crystal axes in the direction normal to the surface.

30

The tilt and twist of interest are typically in the range 0 degrees to 20 degrees, and in particular small angles in the range 0 degrees to 5 degrees. At larger angles, tilt is usually described in terms of the crystal facet index. At larger twist angles, the periodic interface structure is usually described in terms of coincidence site lattices.

The first crystal axis may differ from the second crystal axis by a twist angle  $\theta$ , where  $\theta$  may be within the range 0,1-10°, such as in the range 0,2-9°, such as in the range 0,3-8°, such as in the range 0,5-7°, such as in the range 1-5°.

5 Alternatively or in addition, the first crystal axis may differ from the second crystal axis by a tilt angle  $\varphi$ , where  $\varphi$  may be within the range 0,1-10°, such as in the range 0,2-9°, such as in the range 0,3-8°, such as in the range 0,5-7°, such as in the range 1-5°.

10 In the third aspect, the present invention relates to a laser comprising:

- an article according to the second aspect of the present invention, the article further comprising:

15 - a material or a material system so as to form an array of quantum dots and/or quantum wires at or near the interface region, wherein the further material or material system distributes according to the substantially periodic pattern, and

20 - means for providing a pump signal for pumping the material or material system so as to emit electromagnetic radiation.

The material or material system forming the array of quantum dots and/or quantum wires may comprise a semiconductor material, such as gallium, arsenide or indium or  
25 any combination thereof. Furthermore, the material or material system forming the array of quantum dots and/or quantum wires has a thickness smaller than 200 nm, such as smaller than 150 nm, such as smaller than 100 nm, such as smaller than 80 nm, such as smaller than 50 nm.

30 The material or material system forming the array of quantum dots and/or quantum wires may be overgrown with an additional material, such as silicon or gallium arsenide.



The pump signal may comprise electromagnetic radiation in the radio frequency, visible or near-infrared range. The pump signal may also comprise a direct or alternating electric current passed through the quantum dots to stimulate the emission of radiation. The emitted electromagnetic radiation may be in the visible or near-infrared range.

5

In a fourth aspect, the present invention relates to an object for calibrating an instrument, said object comprising:

- an article according to the second aspect of the present invention, wherein
- 10 the substantially periodic pattern is transferred to a surface of the article.

The transferring of the substantially periodic pattern to the surface may comprise removing at least part of the first or second group of crystalline elements by etching. Alternatively, the transferring of the substantially periodic pattern to the surface may

15 comprise removing at least part of the first or second group of crystalline elements by chemical-mechanical polishing.

The transferred substantially periodic pattern is adapted to hold an additional material, such as a metal.

20

In the fifth aspect, the present invention relates to an element for splitting an incoming beam into one or more outgoing beams, said element comprising:

- an article according to the second aspect of the present invention, wherein
- 25 the incoming beam is incident on at least part of the substantially periodic pattern, said incoming beam having a first propagating direction, and wherein the one or more outgoing beams are reflected or transmitted by at least part of the substantially periodic pattern in one or more propagation directions being different from the first propagation direction.

30

In a sixth aspect, the present invention relates to an object for magnetically storing information, the said object comprising

- an article according to the second aspect of the present invention, wherein the substantially periodic pattern is transferred to a surface of the article, said transferred substantially periodic pattern being adapted to hold a plurality of magnetic structures so as to form a plurality of magnetic domains.

5

The plurality of magnetic structures may comprise iron, cobalt, chromium or any combination thereof. The plurality of magnetic structures are arranged according to the substantially periodic structure. The plurality of magnetic structures may be overgrown with a non-magnetic material.

10

#### BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 illustrates the displacement field near the interface of two crystals bonded at their (001) surfaces at a twist angle  $\theta$ . (A) The bonding produces atomic displacements within a layer of characteristic thickness  $t$ . (B) Grid illustrating the modulation of the atom positions in a layer perpendicular to the interface. (C) The two atomic layers on either side of the interface, illustrating the dislocation network of period  $d$ . The model described in the following section has been used to calculate the atom positions in (B) and (C). The line  $ss$  in (C) shows where the layer in (B) cuts through the interface.

Fig. 2. (A) The reciprocal lattice in a plane parallel to the interface.  $P_1$  and  $P_2$  are bulk Bragg  $(1\bar{1}1)$  reflections and the cross indicate a satellite reflection. (B) The satellite reflection  $S$  in (A) scanned along the in-plane direction  $[h,h,0]$  at  $l = 1$ , for a sample with a twist angle of  $7.45^\circ$ . The full width half maximum of the reflection is  $\Delta h = 1.5 \cdot 10^{-4}$  in reciprocal lattice units (r.l.u.). (C) A series of scans through the satellite reflection  $S$  at  $l = 1$ , measured along the out-of-plane direction  $l$ , for twist angles between  $0.41^\circ$  and  $7.45^\circ$ . The abscissa  $\Delta l$  measures the distance from the satellite point at  $l = 1$ . The solid curves are fitted Lorentzian-squared functions. A measured background intensity has been subtracted from all data points and the individual curves have been shifted vertically for clarity.

Fig. 3. Open circles are the measured full width half maximum values  $w$  for scans along the out-of-plane direction  $l$  (a subset is shown in Fig. 2C) as a function of the

twist angle  $\theta$ . The full line is calculated using the screw dislocation model described in the text. The inset shows the same plot on a linear scale.

Fig. 4 shows the calculated displacements  $dy$  of atoms along the  $x$  direction in the interface plane, given relative to the unperturbed crystal far away from the interface at different layers  $z$  from the interface, for a twist angle of  $\theta = 4.4^\circ$ . The open circles are calculated with the screw dislocation model described in the text. The solid lines are fitted sine functions. The amplitude of the sine function decays exponentially with  $z$ . The curves are shifted vertically for clarity.

10

#### DETAILED DESCRIPTION OF THE INVENTION

Periodic elastic modulation of a semiconductor crystal on the nanometer scale gives rise to novel electronic and optical properties. For example, external elastic  
15 modulation of a buried quantum well can lead to the formation of quantum wires or quantum dots.

Several methods have been developed to introduce a lateral periodic elastic modulation in the subsurface region of a semiconductor: cleaved edge overgrowth on a multiple quantum well, subsurface strain due to self-organised nanoscale islands or surface acoustic waves. Each of these techniques has limitations in either the extent,  
20 regularity or temporal stability of the periodic modulation that can be produced. Alternative methods to achieve lateral periodic elastic modulation extending a significant depth into a semiconductor are therefore of considerable interest.

25

Fusion bonding of two semiconductor wafers, where the orientation of one wafer is twisted with respect to the other by an angle  $\theta$  (see Fig. 1A) results in the formation of a regular network of screw dislocations at the interface (see Fig. 1B,C), as observed by transmission electron microscopy. The lattice spacing  $d$  of the dislocation  
30 network is given by:

$$d = \frac{(a/\sqrt{2})}{2 \sin(\theta/2)} \quad (1)$$

where  $a = 5.43 \text{ \AA}$  is the lattice constant of silicon, and  $a/\sqrt{2}$  is the nearest-neighbor interatomic distance in the (001) interface plane. Such artificial twist grain boundaries have been studied extensively for gold bicrystals. Bonded semiconductor wafers have been studied at large twist angles ( $\theta > 10^\circ$ ), where thin crystals become elastically compliant, indicating that the elastic perturbation of one crystal by the other is small.

The present invention focuses on small twist angles. It is demonstrated that the characteristic thickness  $t$  of the elastic modulation, defined as the sum of the exponential decay lengths of the modulation amplitude to either side of the interface (see Fig. 1), is inversely proportional to the twist angle  $\theta$ .

The technique used to investigate the elastic modulation near the interface is synchrotron X-ray diffraction. The penetration of high intensity X-rays enables the non-destructive structural investigation of buried interfaces. The samples were prepared by direct wafer bonding, without an intermediate adhesive or oxide layer, which is a well-established technique for a wide variety of applications in microelectronics and micromechanics. The wafers used were 10 cm diameter commercial grade mirror-polished Si(001), 350  $\mu\text{m}$  thick, and were stripped of their native oxide in a 5% HF solution prior to contacting in a class 100 clean-room. The contacted pairs were then annealed at 1000°C for 1 hr in a nitrogen atmosphere to achieve high-strength silicon covalent bonding at the interface. Samples ( $\sim 1 \text{ cm}^2$ ) diced from wafer pairs that were bonded with twist angles between 0.4° and 7.5°. The tilt angle (miscut) of the wafers, defined as the misorientation of the physical surface relative to the crystallographic [001] direction, was less than 0.1°. To reduce X-ray absorption, one of the two bonded wafers was thinned to about 30  $\mu\text{m}$  by mechanical grinding followed by chemical etching. The X-ray measurements were performed at the undulator beamline ID32 at the European Synchrotron Radiation Facility (ESRF) and at the wiggler beamline BW2 at HASYLAB, using six circle diffractometers.

The wavelengths used were 0.5254  $\text{\AA}$  and 1.24  $\text{\AA}$  at ID32 and HASYLAB, respectively. Most of the experiments were performed at ID32, only one sample, where one of the wafers was thinned to 1.5  $\mu\text{m}$  was measured at BW2. The typical beam size

at sample at ID32 was 0.5mm vertical, 0.2mm horizontal. The samples were mounted in air with sample surface normal aligned with the  $\omega$  - rotation axis. The angle  $\alpha$  between incoming x-ray beam and sample surface was kept constant during scans, typically  $\alpha = 3^\circ$ , giving a typical exit angle  $\beta = 2.5^\circ$ . For these angles and the  
 5 quoted sample thickness, the total beam attenuation is about 30%. The detector slits before the sample was typically  $0.5 \times 0.5 \text{ mm}^2$ . The intensities along satellite rods were measured by measuring the peak and subtracting the average background from either side of the peak. Several rods were measured using the conventional  $\omega$ -  
 10 background were measured more simply by scanning parallel to the rod.

A superstructure of period,  $d$ , confined to a region near the bonded interface results in an array of Bragg reflections in the reciprocal space probed by X-ray diffraction. These reflections are elongated in the direction normal to the interface and spaced by  
 15  $2\pi/d$  in the plane of the interface. For a nearly harmonic displacement of the atomic positions, only the satellite reflections closest to the bulk Bragg reflections of the two crystals (see Fig. 2A) have significant intensity. Conventional cubic reciprocal lattice notation is used. The width  $\Delta h$  of these satellite reflections along the in-plane  $[h,h,0]$  direction in reciprocal space is inversely proportional to the lateral coherence length  $\xi$   
 20 of the periodic superstructure,  $\xi = (a/\sqrt{2})/\Delta h$ .

Fig. 2B shows a scan through a satellite reflection  $S$  close to the  $(1\bar{1}1)$  Bragg reflection of a bonded wafer pair with  $\theta = 7.45^\circ$ . The peak width is  $\Delta h = 1.5 \times 10^{-4}$  in reciprocal lattice units and is resolution limited. From this value a lower limit to the coherence length of  $\xi > 2\mu\text{m}$  is deduced. This is much greater than the typical terrace  
 25 widths on a free surface, which are  $\sim 1000 \text{ \AA}$  for a miscut of  $0.1^\circ$ . This implies that the long-range order of the dislocation network is determined by the perfection of the bulk crystals rather than their surfaces.

30 The satellite reflections closest to the  $(1\bar{1}1)$  reflections were scanned along the out-of-plane direction for a set of seven bonded wafer pairs with different twist angles. Four examples are shown in Fig. 2C. The line shape of the measured reflections is fitted with a Lorentzian-squared function:

$$I(\Delta l) = I_0 \left( \frac{b^2}{b^2 + \Delta l^2} \right)^2 \quad (2)$$

5

where the full width at half maximum is  $w = 2b\sqrt{\sqrt{2}-1}$  and  $\Delta l$  is the distance to the satellite point at  $l = 1$ . For example, for  $\theta = 0.41^\circ$ , a fit to the data in Fig. 2C of a Lorentzian line shape with  $p$ , the power of the Lorentzian, as a fit parameter, gives  $p = 2.07 \pm 0.10$ . A Lorentzian-squared function is the Fourier transform of a double-sided exponential decay function, in other words the elastic modulation decays exponentially to both sides of the interface. The characteristic thickness  $t$  is related to  $w$  by  $t = \frac{a}{\pi b}$ . Thus, as the characteristic thickness  $t$  of the periodically modulated region increases the satellite Bragg peaks sharpen along the  $l$ -direction, as shown in Fig. 2C.

15 In Fig. 3, the measured width of these peaks is plotted against the twist angle  $\theta$ , which can be accurately measured from the angular separation of the bulk Bragg peaks of the two crystals. A linear relationship between  $w$  and  $\theta$  is observed, which implies that  $t$  diverges as  $\theta$  tends to zero.

20 The results are summarized in table 1 where the double exponential decay length  $t$  of the displacement field, as derived from the measured diffraction profiles perpendicular to the interface, is listed. The full width half maximum  $w$  is measured in reciprocal lattice units. The twist angle  $\theta$  and period  $d$  are shown in Fig. 1.

$\theta$	$d$ (Å)	$w$ (r.l.u.)	$t$ (Å)
$0.41^\circ \pm 0.05^\circ$	230	$0.014 \pm 0.001$	159
$1.34^\circ \pm 0.07^\circ$	164	$0.039 \pm 0.003$	58
$1.40^\circ \pm 0.12^\circ$	157	$0.050 \pm 0.005$	45
$3.76^\circ \pm 0.10^\circ$	58	$0.147 \pm 0.010$	14
$4.41^\circ \pm 0.10^\circ$	50	$0.20 \pm 0.02$	11
$5.74^\circ \pm 0.10^\circ$	38	$0.19 \pm 0.02$	12
$7.45^\circ \pm 0.15^\circ$	29	$0.34 \pm 0.02$	6.5

Table 1

In order to interpret these results quantitatively, the displacement field has been simulated numerically for two simple cubic lattices, forming a square network of dislocations at their common interface. Using an analytical expression for the displacement field of a screw dislocation in a cubic lattice, the atomic displacement pattern due to a square array of screw dislocations spaced by  $N = d/a$  lattice parameters have been calculated. Assuming the crystal is elastically isotropic, the result is independent of the specific elastic constants of the crystal.

Fig. 4 shows the lateral modulation,  $\Delta y$ , of the atomic positions along the in-plane  $x$ -direction of one of the cubic lattices for  $N = 13$  corresponding to  $\theta = 4.4^\circ$ , for different atomic layers at distance  $z$  from the interface.

15

In the simulations, the amplitude of the modulation of atomic positions decays exponentially with depth from the interface over several decades, in agreement with experiment. Further, the characteristic thickness  $t$  deduced from these simulations, when plotted as a corresponding peak width, agrees quantitatively with measurements (full line in Fig. 3), confirming the validity of this simple model. For Eqn. 1 in the small angle limit,  $d$  is inversely proportional to  $\theta$ , and so proportional to  $t$ . Although one might expect a higher density of screw dislocations to lead to larger displacements of atoms from their equilibrium positions and hence a larger depth of the modulation, in fact the opposite is observed. This can be understood qualitatively by noting that, at points between two parallel screw dislocations, the displacement field due to each screw dislocation has opposite sign and tends therefore to cancel

25

out. Thus at large twist angles, the effect of the dislocation network is strongly localized, and long-range elastic interaction between the crystal lattices is suppressed, consistent with the behavior of compliant substrates bonded at large twist angles.

5

In contrast, at small twist angles, the elastic modulation can be made to extend many nanometers into the silicon wafers. Indeed, the modulation penetrates much further into the crystals than the characteristic thickness  $t$ , due to the long exponential tails.

10

This effect could be used to modulate a buried quantum well in a semiconductor, resulting in a highly ordered quantum dot structure. The dominant component of the strain at the interface is shear strain. For a twist angle of  $\theta = 5^\circ$  and at layer  $z = 5$ , a maximum shear strain of 1.5% is obtained from the above model. This is comparable to the strain amplitudes estimated for other modulation mechanisms. The elastically modulated region provides a regular lattice with periodicity in the range 1-10 nm, a range for metrology applications that is difficult to access with lithographic techniques. In this case, the modulated region could be exposed by etching to within a few nanometers of the interface. This modulated substrate could also be used as a template for overgrowth of periodically strained layers.

It has been demonstrated that for a series of bonded Si(001) wafers with twist angles ranging from  $0.4^\circ$  to  $7.5^\circ$  the atomic displacement field decays exponentially away from the interface with a characteristic length inversely proportional to the twist angle. The strong diffraction signal observed at small twist angles from the superstructure of the crystals allows a detailed line shape analysis of the measured rod profiles. The data strongly support a simple model for the interface structure, where the atomic displacements are determined by a square net of screw dislocations.

30 The results described in the present text are results for silicon (001) with a twist boundary. However, as the simulations show, the elastic modulation due to a dislocation array is not sensitive to the detailed elastic properties of the material involved, but depends primarily on the spacing of the dislocations. A wide variety of materials can be successfully fusion bonded, including semiconductors such as GaAs, and



insulators such as diamond and sapphire. Also, tilt boundaries will result in parallel rows of edge dislocations, with analogous consequences.

Thus, the results presented here can be generalized to a wide range of materials and crystallographic interfaces. Determination of atomic positions in the core of the dislocations require accurate form factor measurements including many higher order reflections which go beyond the present analysis, but is quite feasible for future studies.

- 10 In a first embodiment the periodic strain field may be used as a template for a metrological calibration standard for Scanning Tunnelling Microscopy (STM), Atomic Force Microscopy (AFM) and Scanning Electron Microscopy (SEM) in the 3-100 nm regime. Calibration standards in this regime are difficult to obtain; the lattice constant of standard crystalline materials are too small and most artificial structures have too
- 15 large a lattice constant. A calibration standard can be accomplished by wafer bonding two Si(100) wafers with zero tilt at twist angles of  $\theta$ , where  $\theta$  is in the range  $0.2^\circ$ - $7^\circ$ . This results in a periodic dislocation network with lattice spacing given by equation (1).
- 20 In order to be used as a calibration standard this periodic lattice must be transferred to the surface of one of the two crystals. After bonding, one of the two crystals must be thinned down to 100 nm or less, in order to allow the periodic strain field in the direction perpendicular to the bonded interface to extend to the surface of the thinned crystal. The thinning can be achieved by preparing one of the wafers with an
- 25 H- implanted etch stop prior to bonding, and etching with standard etchants such as KOH to remove the silicon on one side of the interface down to the etch stop. As shown in table 1 the strain field has an exponential decay away from the interfaces with  $1/e$  lengths of 1-30 nm, so the etch stop must have a comparable depth. Alternatively, chemical-mechanical polishing techniques could be applied to thin one
- 30 of the wafers. Alternatively, thin membranes could be produced in one of the wafers prior to bonding, by chemical or physical etching techniques such as reactive ion etching. Roughness of the surface defined by the etch stop due to etching can be reduced, for example, by repeated oxidation and etching in HF.

When the periodic strain field penetrates to the surface, it can be made more easily detectable, for example by overgrowth of a suitable material, such as a metal. A material is chosen which shows preferential growth at sites with a specific strain, leading to a replication of the periodic pattern of the strain field in the overgrown material. A metallic overlayer would also have advantages for STM measurements, where the substrate must be conducting. The pattern will have the same periodicity as the underlying grain boundary, and can be imaged with the above mentioned scanning techniques. The period of the pattern is given by the twist angle between the two crystals as described in equation (1), and can be measured independently and extremely accurately by, for example, X-ray techniques, in order to provide a standard reference for the scanning probe techniques. The twist angle can be determined with accuracy better than  $0.01^\circ$ . A single bonded wafer could produce hundreds of identical calibration standards for distribution by manufacturers of scanning probe systems to their customers. Owing to the two dimensional nature of the periodic strain field, the calibration standards could also be used for calibrating distortions that affect the relative scales of perpendicular axes in the scanned images.

In a second embodiment the periodic strain field may be used as a template for growth of quantum dot lasers. The efficiency of such lasers depend on being able to produce a very high density of clusters of semiconductor material on a surface, the cluster size being in the nanometer range, and the clusters being nearly monodisperse. The optical properties of such clusters can be compared to artificial atoms with sharp absorption lines, and this has a number of advantages for laser fabrication in, for example, telecommunications applications. In particular, owing to the line spectra of such quantum dots, the wavelength of spontaneous emission of the dots is much more stable to temperature changes than so-called quantum wire or quantum well lasers. However, a key challenge in the fabrication of such dots by self organisation techniques has been the compromise between achieving a dense packing, and preventing clustering.

Producing a periodic strain modulation at a free surface provides a way of controlling the cluster formation process by providing a periodic array of preferred nucleation sites as a template for overgrowth of quantum dots. The periodic lattice must be

transferred to the surface of one of the two crystals by the method described in the previous embodiment.

After bonding, one of the two crystals must be thinned down to about 100 nm, in order to allow the strain field in the direction perpendicular to the bonded interface to extend to the surface of the thinned crystal. The thinning can be achieved by preparing one of the wafers with an H- implanted etch stop prior to bonding, and etching with standard etchants such as KOH to remove the silicon on one side of the interface down to the etch stop. As shown in table 1 the strain field has an exponential decay away from the interfaces with  $1/e$  lengths of 1-30nm, so the etch stop must have a comparable depth. Roughness of the surface defined by the etch stop due to etching can be reduced, for example, by repeated oxidation and etching in HF.

When the periodic strain field penetrates to the surface, any oxide or contamination layer at this surface can be removed in vacuum by standard techniques such as high-temperature annealing and sputtering. Subsequently, deposition of semiconductor materials suitable for quantum dots, such as Indium Arsenide can be carried out, in a molecular beam epitaxy system or chemical vapour deposition system. Under suitable conditions of substrate temperature and deposition rate, the clusters will grow preferentially at specific sites on the strained surface, which minimise the strain energy between the overgrown quantum dots and the substrate. In this way, a periodic pattern of quantum dots can be made. By varying the twist angle of the semiconductor wafers prior to bonding, the spacing of the quantum dots can be controlled, to achieve optimum density for a given mean quantum dot size. By varying the material the wafers are made of (e.g. silicon, gallium arsenide) the electronic state of the quantum dots, and hence their optical properties, can be controlled. After preparation of the quantum dots, a protective cap layer of semiconductor material can be deposited on the quantum dots to protect them from oxidation. Further electrical connections to provide injection of carriers can be fabricated by prior art methods. Injection of charge carriers is necessary to stimulate lasing.

- In a third embodiment the periodic strain field may be used as a modulator of a buried quantum well layer, in order to form a quantum dot laser (described above). In this embodiment, it is not necessary to thin one of the wafers very accurately. Instead, prior to bonding, a quantum well structure is fabricated in one of the wafers,
- 5 with a suitable thin protective layer. Such a quantum well structure consists of a nanometer-thickness layer of one type of semiconductor grown epitaxially on another, for example gallium indium arsenide on gallium arsenide, using standard techniques such as molecular beam epitaxy. The thickness of the thin protective layer is 100nm or less, such that the periodic strain field generated at the interface
- 10 between the two wafers can penetrate through the protective layer and modulate the quantum well. This strain modulation will have the effect of modulating the electronic band-structure of the charge carriers in the, creating periodic electronic structure analogous to an array of quantum dots.
- 15 In prior art, such sub-surface modulation has been produced by growth of clusters at a free surface. This embodiment provides a more controlled technique, where by varying the twist angle of the semiconductor wafers prior to bonding, the spacing of the quantum dots can be controlled, to achieve optimum density for a given mean quantum dot size. After preparation, further electrical connections to the modulated
- 20 quantum well can be fabricated by prior art. These provide a means to inject charge carriers in order to stimulate lasing.

- In a fourth embodiment the periodic strain field may be used as a template for growth of arrays of periodic magnetic nanostructures, for applications such as data
- 25 storage. An important challenge in data storage materials is the fabrication of regular magnetic nanodomains that can be addressed individually. Self-organisation techniques for achieving this depend on a compromise between achieving a dense packing, and preventing clustering.
- 30 Producing a periodic strain modulation at a free surface provides a way of controlling the cluster formation process by providing a periodic array of preferred nucleation sites as a template for overgrowth of magnetic nanostructures. The periodic lattice must be transferred to the surface of one of the two crystals by the method described in the first embodiment.

Again, after bonding, one of the two crystals must be thinned down to about 100 nm, in order to allow the strain field in the direction perpendicular to the bonded interface to extend to the surface of the thinned crystal. The thinning can be  
5 achieved by preparing one of the wafers with an H- implanted etch stop prior to bonding, and etching with standard etchants such as KOH to remove the silicon on one side of the interface down to the etch stop. As shown in table the strain field has an exponential decay away from the interfaces with  $1/e$  lengths of 1-30nm, so the etch stop must have a comparable depth. Roughness of the surface defined by  
10 the etch stop due to etching can be reduced, for example, by repeated oxidation and etching in HF.

When the periodic strain field penetrates to the surface, any oxide or contamination layer at this surface can be removed in vacuum by standard techniques such as high-  
15 temperature annealing and sputtering. Subsequently, magnetic materials are deposited suitable for data storage, such as iron, cobalt, chromium, and alloys of such materials, using techniques such as molecular beam epitaxy system or chemical vapour deposition system. Under suitable conditions of substrate temperature and deposition rate, the clusters will grow preferentially at specific sites on the strained  
20 surface, which minimise the strain energy between the overgrown magnetic nanostructures and the substrate. Optionally, a buffer layer of one metallic material may be deposited prior to the active magnetic material, in order to optimise diffusion of the magnetic material. In this way, a periodic magnetic pattern can be made. By varying the twist angle of the semiconductor wafers prior to bonding, the spacing of  
25 the quantum dots can be controlled, to achieve optimum density for a given mean quantum dot size.

## CLAIMS

1. A method for providing a substantially periodic pattern, said method comprising the steps of:

5

- providing a first group of crystalline elements being formed by the same material and having a predetermined first crystal axis,

10

- providing a second group of crystalline elements being formed by the same material and having a predetermined second crystal axis being different from the first crystal axis, and

- bringing the first and second group of crystalline elements into contact with each other.

15

2. A method according to claim 1, wherein the material forming the first group of crystalline elements comprises a semiconductor material, such as silicon or gallium arsenide.

20

3. A method according to claim 1 or 2, wherein the material forming the second group of crystalline elements comprises a semiconductor material, such as silicon or gallium arsenide.

25

4. A method according to claim 1, wherein the materials forming the first and second group of crystalline elements comprise an insulator material, such as diamond or sapphire.

30

5. A method according to any of claims 1-4, wherein the first crystal axis differs from the second crystal axis by a twist angle  $\theta$ , wherein  $\theta$  is within the range 0,1-10°, such as in the range 0,2-9°, such as in the range 0,3-8°, such as in the range 0,5-7°, such as in the range 1-5°.

6. A method according to any of claims 1-4, wherein the first crystal axis differs from the second crystal axis by a tilt angle  $\varphi$ , wherein  $\varphi$  is within the range 0,1-10°,

such as in the range 0,2-9°, such as in the range 0,3-8°, such as in the range 0,5-7°, such as in the range 1-5°.

7. An article comprising:

5

- a first group of crystalline elements being formed by the same material and having a predetermined first crystal axis,

10

- a second group of crystalline elements being formed by the same material and having a predetermined second crystal axis being different from the first crystal axis,

15

wherein the first and second group of crystalline elements are adjacently positioned so as to form an interface region between the first and second group of crystalline elements, at least part of said interface region defining a substantially periodic pattern extending in at least one direction.

20

8. An article according to claim 7, wherein the material forming the first group of crystalline elements comprises a semiconductor material, such as silicon or gallium arsenide.

25

9. An article according to claim 7 or 8, wherein the material forming the second group of crystalline elements comprises a semiconductor material, such as silicon or gallium arsenide.

30

10. An article according to claim 7, wherein the materials forming the first and second group of crystalline elements comprise an insulator material, such as diamond or sapphire.

11. An article according to any of claims 7-10, wherein the first crystal axis differs from the second crystal axis by a twist angle  $\theta$ , wherein  $\theta$  is within the range 0,1-10°, such as in the range 0,2-9°, such as in the range 0,3-8°, such as in the range 0,5-7°, such as in the range 1-5°.

12. An article according to any of claims 7-10, wherein the first crystal axis differs from the second crystal axis by a tilt angle  $\phi$ , wherein  $\phi$  is within the range 0,1-10°, such as in the range 0,2-9°, such as in the range 0,3-8°, such as in the range 0,5-7°, such as in the range 1-5°.

5

13. A laser comprising:

- an article according to any of claims 7-12, the article further comprising:

10

- a material or a material system so as to form an array of quantum dots and/or quantum wires at or near the interface region, wherein the further material or material system distributes according to the substantially periodic pattern, and

15

- means for providing a pump signal for pumping the material or material system so as to emit electromagnetic radiation.

14. A laser according to claim 13, wherein the material or material system forming the array of quantum dots and/or quantum wires comprises a semiconductor

20

material, such as gallium, arsenide or indium or any combination thereof.

15. A laser according to claim 13 or 14, wherein the material or material system forming the array of quantum dots and/or quantum wires has a thickness smaller than 200 nm, such as smaller than 150 nm, such as smaller than 100 nm, such as

25

smaller than 80 nm, such as smaller than 50 nm.

16. A laser according to any of claims 13-15, wherein the material or material system forming the array of quantum dots and/or quantum wires is overgrown with an additional material, such as silicon or gallium arsenide.

30

17. A laser according to any of claims 13-16, wherein the pump signal comprises electromagnetic radiation.



18. A laser according to claim 17, wherein the electromagnetic radiation is in the radio frequency, visible or near-infrared range.
19. A laser according to any of claims 13-16, wherein the pump signal comprises a  
5 direct and/or alternating electric current.
20. A laser according to any of claims 13-19, wherein the emitted electromagnetic radiation is in the visible or near-infrared range.
- 10 21. An object for calibrating an instrument, said object comprising:
- an article according to any of the claims 7-12, wherein the substantially periodic pattern is transferred to a surface of the article.
- 15 22. An object according to claim 21, wherein the transferring of the substantially periodic pattern to the surface comprises removing at least part of the first or second group of crystalline elements by etching.
23. An object according to claim 21, wherein the transferring of the substantially periodic pattern to the surface comprises removing at least part of the first or second  
20 group of crystalline elements by chemical-mechanical polishing.
24. An object according to claim 21, wherein the transferred substantially periodic pattern is adapted to hold an additional material, such as a metal.
- 25
25. An element for splitting an incoming beam into one or more outgoing beams, said element comprising:
- an article according to any of the claims 7-12, wherein the incoming beam is  
30 incident on at least part of the substantially periodic pattern, said incoming beam having a first propagating direction, and wherein the one or more outgoing beams are reflected or transmitted by at least part of the substantially periodic pattern in one or more propagation directions being different from the first propagation direction.

26. An object for magnetically storing information, the said object comprising

- 5       - an article according to any of the claims 7-12, wherein the substantially periodic pattern is transferred to a surface of the article, said transferred substantially periodic pattern being adapted to hold a plurality of magnetic structures so as to form a plurality of magnetic domains.

27. An object according to claim 26, wherein the plurality of magnetic structures  
10       comprise iron, cobalt, chromium or any combination thereof.

28. An object according to claim 26 or 27, wherein the plurality of magnetic structures are arranged according to the substantially periodic structure.

15       29. An object according to any of claims 26-28, wherein the plurality of magnetic structures are overgrown with a non-magnetic material.

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization  
International Bureau



(43) International Publication Date  
4 January 2001 (04.01.2001)

PCT

(10) International Publication Number  
**WO 01/00522 A2**

- (51) International Patent Classification<sup>7</sup>: **B81B 1/00**
- (21) International Application Number: **PCT/DK00/00348**
- (22) International Filing Date: **28 June 2000 (28.06.2000)**
- (25) Filing Language: **English**
- (26) Publication Language: **English**
- (30) Priority Data:  
PA 1999 00918 **28 June 1999 (28.06.1999)** **DK**
- (71) Applicant (*for all designated States except US*):  
**MIKROELEKTRONIK CENTRET (MIC)** [DK/DK];  
The Technical University of Denmark (DTU), Building  
345 East, DK-2800 Lyngby (DK).
- (72) Inventors; and
- (75) Inventors/Applicants (*for US only*): **GREY, Hasin,**  
**François de Charmoy** [CA/DK]; Boldhusgade 4, 1,  
DK-1602 Copenhagen K (DK). **FEIDENHANS'L,**  
**Robert, Krarup** [DK/DK]; Møllehusvej 64, DK-4000  
Roskilde (DK). **VEDDE, Jan** [DK/DK]; J.N. Vinthersvej  
5, DK-3460 Birkerød (DK). **NIELSEN, Mourits**  
[DK/DK]; Nakskovvej 14, Veddelev, DK-4000 Roskilde  
(DK). **HOWES, Paul, Bedford** [GB/GB]; 6 Dean Road,  
Hinckley, Leicestershire LE10 1LG (GB).
- (54) Title: **NANOMETER-SCALE MODULATION**
- (57) Abstract: A new method to artificially modulate a crystal lattice is disclosed, where the modulation has a controlled periodicity and thickness in the range of one to several hundred nanometers. The present invention relates to the fabrication of periodically strained crystal lattices where the period and thickness of the modulated region is controlled by bonding two crystal wafers at a specified twist angle. Two polished and clean crystal wafers are placed in intimate contact at a specified twist angle and, if necessary, heated to obtain bonding between the two wafers. The two crystal lattices modulate each other, resulting in a modulation near the interface between the crystal with a periodicity that is different from that of the crystal lattices. This periodic modulation affects the electronic and structural properties of the two crystals in the vicinity of the interface. The modulation of the electronic properties leads to the formation of a highly regular quantum dot or quantum wire structures of controlled period near the crystal interface, with applications in electronics, optoelectronics and magnetic devices. The modulation of the crystal structure can be exploited for the formation of metrological standards in the 1-100 nanometer range, or gratings for diffractive optic elements with grating periods in the same range. By transferring the periodic modulation to the free surface of a crystal, it can be used as a template for further growth of periodically modulated crystalline layers by evaporation techniques.
- (74) Agent: **PLOUGMANN, VINGTOFT & PARTNERS**  
A/S; Sankt Annæ Plads 11, P.O. Box 3007, DK-1021  
Copenhagen K (DK).
- (81) Designated States (*national*): AE, AG, AL, AM, AT, AT  
(utility model), AU, AZ, BA, BB, BG, BR, BY, CA, CH,  
CN, CR, CU, CZ, CZ (utility model), DE, DE (utility  
model), DK, DK (utility model), DM, DZ, EE, EE (utility  
model), ES, FI, FI (utility model), GB, GD, GE, GH, GM,  
HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KR (utility  
model), KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG,  
MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE,  
SG, SI, SK, SK (utility model), SL, TJ, TM, TR, TT, TZ,  
UA, UG, US, UZ, VN, YU, ZA, ZW.
- (84) Designated States (*regional*): ARIPO patent (GH, GM,  
KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian  
patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European  
patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE,  
IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG,  
CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).
- Published:  
— *Without international search report and to be republished  
upon receipt of that report.*
- For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.*

WO 01/00522 A2

1/4

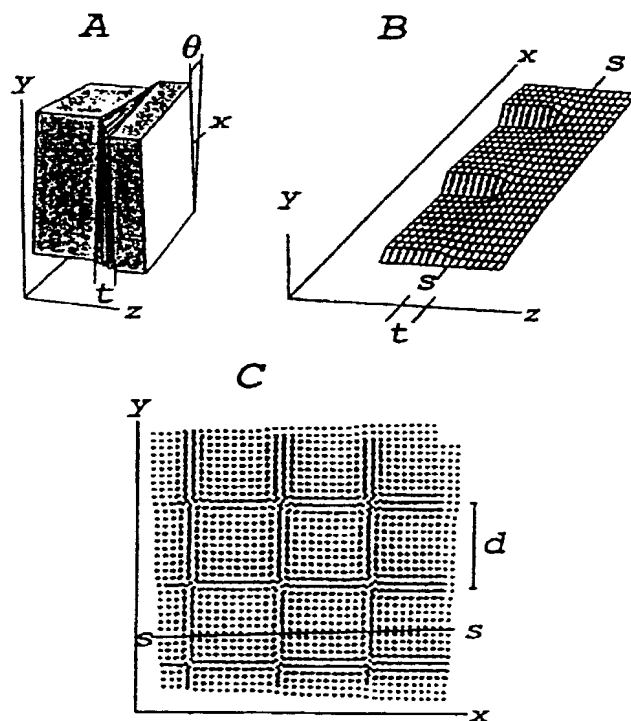


Fig. 1

2/4

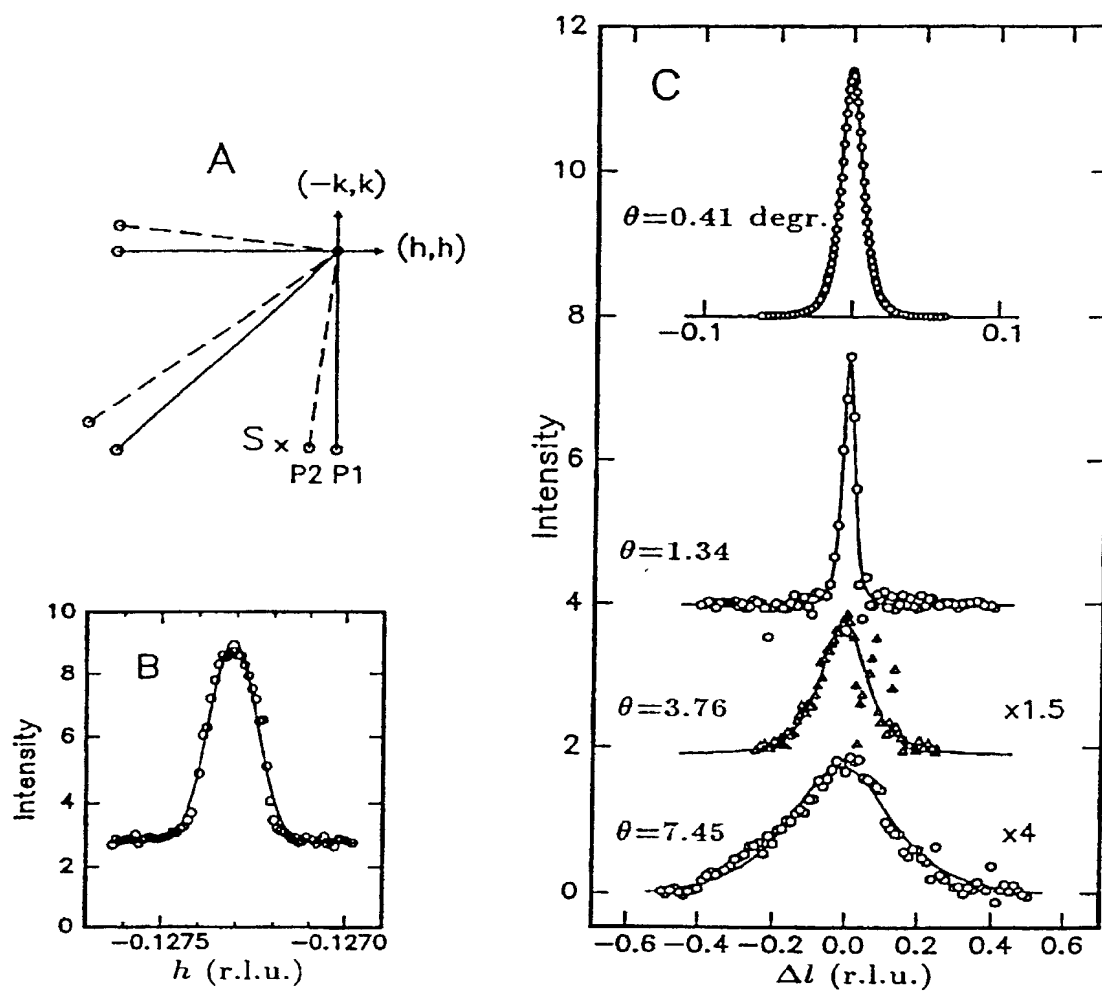


Fig. 2

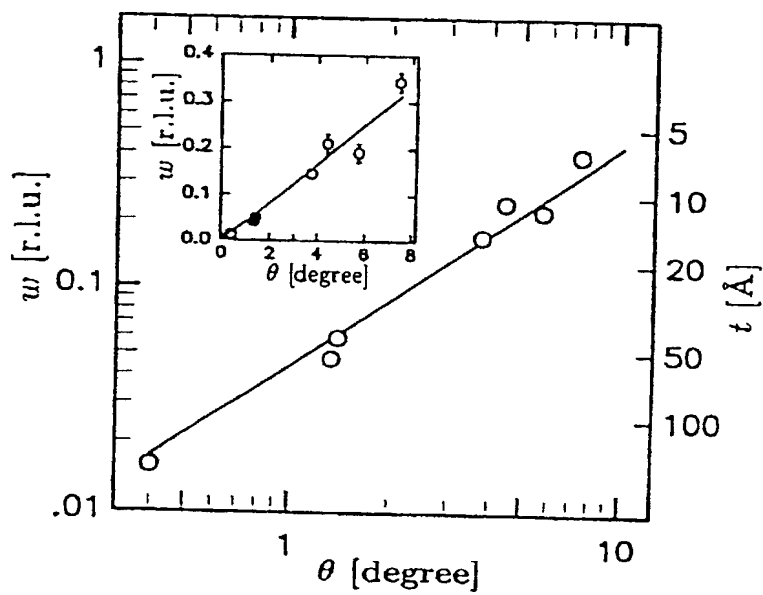


Fig. 3

4/4

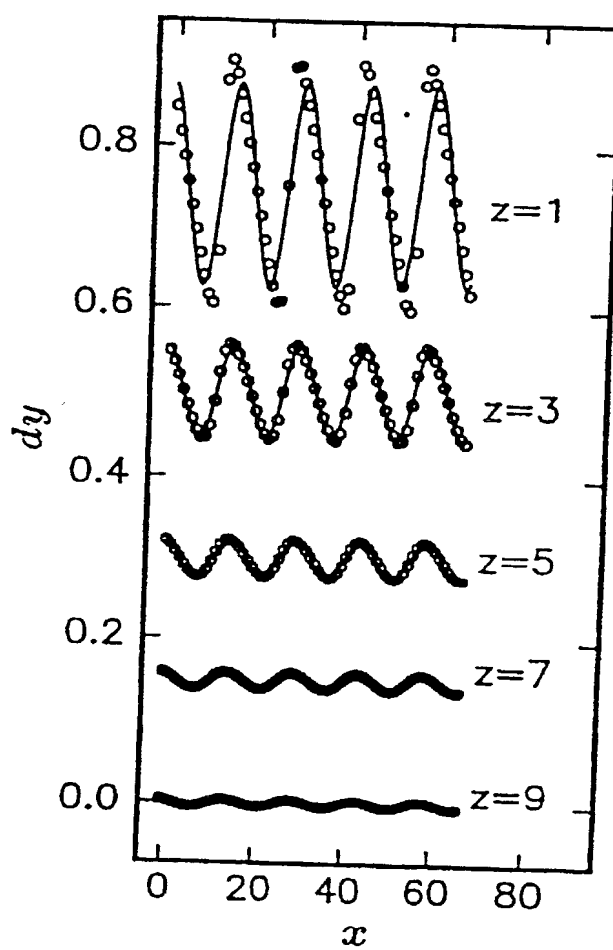


Fig. 4

# DECLARATION AND POWER OF ATTORNEY

#5

Atty. Dkt. No.: 45900-000714/US

## DECLARATION

As a below named inventor, I hereby declare that:

My residence, mailing address and citizenship are as stated below next to my name,

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

### NANOMETER-SCALE MODULATION

the specification of which (check one)

- ☐ is attached hereto.  
or  
☒ was filed on June 28, 2000 as PCT International Application No. PCT/DK00/00348 and was amended on \_\_\_\_\_ (if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR § 1.56, including for continuation-in-part applications, material information which became available between the filing date of the prior application and the national or PCT international filing date of the continuation-in-part application.

I hereby claim foreign priority benefits under 35 U.S.C. §§ 119(a)-(d) or 365(b) of any foreign application(s) for patent or inventor's certificate, or 365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or any PCT international application having a filing date before that of the application on which priority is claimed:

PRIOR FOREIGN APPLICATION(S)				
APPN. SERIAL NO.	COUNTRY	DATE FILED (MM/DD/YYYY)	PRIORITY CLAIM	
			Yes	No
PA 1999 00918	DK	06/28/1999	<input checked="" type="checkbox"/>	<input type="checkbox"/>
			<input type="checkbox"/>	<input type="checkbox"/>
			<input type="checkbox"/>	<input type="checkbox"/>



## DECLARATION AND POWER OF ATTORNEY

I hereby claim the benefit under 35 U.S.C. § 119(e) of any United States provisional application(s) listed below:

PRIOR PROVISIONAL APPLICATION(S)	
APPN. SERIAL NO.	DATE FILED (MM/DD/YYYY)

I hereby claim the benefit under 35 U.S.C. § 120 of any United States application(s) listed below:

PRIOR U.S. APPLICATION(S)		
APPN. SERIAL NO.	DATE FILED (MM/DD/YYYY)	STATUS - PATENTED, PENDING, ABANDONED

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. § 1001 and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

### POWER OF ATTORNEY

I hereby appoint the following attorneys with full power of substitution and revocation, to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith.

⑥

Thomas S. Auchterlonie	Reg. No. <u>37,275</u>
John A. Castellano	Reg. No. <u>35,094</u>
Terry L. Clark	Reg. No. <u>32,644</u>
Donald J. Daley	Reg. No. <u>34,313</u>
Gary D. Yacura	Reg. No. <u>35,416</u>
Timothy R. Wyckoff	Reg. No. <u>46,175</u>

# DECLARATION AND POWER OF ATTORNEY

## CORRESPONDENCE ADDRESS

I request the Patent and Trademark Office to direct all correspondence and telephone calls relative to this application to Harness, Dickey & Pierce, P.L.C., Customer No. 30593, P.O. Box 8910, Reston, Virginia, 20195, (703) 390-3030.

1-00 Hasin Francois de Charmoy GREY:

Inventor's signature: Hasin Francois de Charmoy

Date: 19 April 2002

Residence: Boldhusgade 4, 1 DK-1602, Copenhagen K, Denmark DKX

Citizenship: Canada

Mailing Address: Boldhusgade 4, 1 DK-1602, Copenhagen K, Denmark

2-00 Robert Krarup FEIDENHANS'L:

Inventor's signature: Robert Krarup

Date: 19/4-02

Residence: Møllehusvej 64, DK-4000, Rodkilde, Denmark DKX

Citizenship: Denmark

Mailing Address: Møllehusvej 64, DK-4000, Rodkilde, Denmark

3-00 Jan VEDDE:

Inventor's signature: Jan Vedde

Date: 19/4 - 2002

Residence: J.N. Vinthersvej 5, DK-3460 Birkerød, Denmark DKX

Citizenship: Denmark

Mailing Address: J.N. Vinthersvej 5, DK-3460 Birkerød, Denmark

DECLARATION AND POWER OF ATTORNEY

4-00 **Mourits NIELSEN:**

Inventor's signature: Mourits Nielsen

Date: 07 05 2002

Residence: Nakskovvej 14, Veddelev, DK-4000, Roskilde, Denmark DKX

Citizenship: Denmark

Mailing Address: Nakskovvej 14, Veddelev, DK-4000, Roskilde, Denmark

**Paul Bedford HOWES:**

Inventor's signature: \_\_\_\_\_

Date: \_\_\_\_\_

Residence: 6 Dean Road, Hinckley, Leicestershire LE10 1LG, Great Britain

Citizenship: Great Britain

Mailing Address: 6 Dean Road, Hinckley, Leicestershire LE10 1LG, Great Britain

**DECLARATION AND POWER OF ATTORNEY****Mourits NIELSEN:**

Inventor's signature: \_\_\_\_\_

Date: \_\_\_\_\_

Residence: Nakskovvej 14, Veddelev, DK-4000, Roskilde, Denmark

Citizenship: Denmark

Mailing Address: Nakskovvej 14, Veddelev, DK-4000, Roskilde, Denmark

5-00 **Paul Bedford HOWES:**Inventor's signature: P. B. HowesDate: 7/5/2002Residence: 6 Dean Road, Hinckley, Leicestershire LE10 1LG, Great Britain GBN

Citizenship: Great Britain

Mailing Address: 6 Dean Road, Hinckley, Leicestershire LE10 1LG, Great Britain